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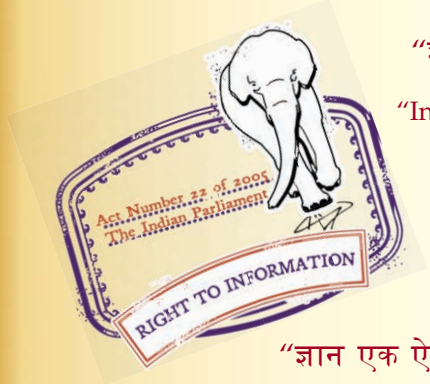

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SPECIFICATION FOR LIQUID AMINE SALTS OF MCPA

UDC 632·954 : 547·586·2

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MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

March 1977

Indian Standard

SPECIFICATION FOR LIQUID AMINE SALTS OF MCPA

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AMENDMENT NO. 1 MAY 1994
TO
IS 8294 : 1976 SPECIFICATION FOR LIQUID AMINE SALTS OF MCPA

(*Page 5, clause* 4.1) -Substitute the following for the existing:

'When freshly manufactured material in bulk quantity is offered for inspection, representative samples of the material shall be drawn and tested as prescribed in IS 10627 : 1983 within 90 days of its manufacture. When the material is offered for inspection after 90 day of its manufacture. sampling shall be done as prescribed in KS 10627 : 1983. However, the criteria for conformity of the material when tested, shall be the limits of tolerances, as applicable over the declared nominal value and given under clause **2.2.1** of the standard.'

(FAD 1)

Reprography Unit, BIS, New Delhi, India

Indian Standard

SPECIFICATION FOR LIQUID AMINE SALTS OF MCPA

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 31 December 1976, after the draft finalized by the Pest Control Sectional Committee had been approved by the Agricultural and Food Products Division Council and the Chemical Division Council.

0.2 Formulations of amine salts of 4-chloro-2-methylphenoxy acetic acid (MCPA) are used extensively in the control of weeds in cereal crops, grassland and turf.

0.3 In the preparation of this standard, due consideration has been given to the provisions of the Insecticides Act, 1968 and the Rules framed thereunder. However, this standard is subject to the restrictions imposed under these, wherever applicable.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for liquid amine salts of 4-chloro-2-methylphenoxy acetic acid (MCPA).

2. REQUIREMENTS

2.1 Description — The product shall consist of an aqueous solution containing MCPA potassium, sodium or amine salts, including mixtures, as the only active ingredients, together with any necessary formulants. The material shall be free from visible suspended matter and sediment.

2.2 The material shall also comply with the requirements given in Table 1.

*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR LIQUID AMINE SALTS OF MCPA

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Method of Test, Ref to Appendix
(1)	(2)	(3)	(4)
i)	Extractable acids including MCPA (expressed as 4-chloro-2-methyl-phenoxy acetic acid), percent by mass	40	A
ii)	Free phenols (expressed as 4-chloro-2-methylphenol), percent by mass, *Max*	1·5	B
iii)	Coarse material, water insoluble percent by mass, *Max*	0·1	C
iv)	Hydrogen ion concentration (*p*H)	7·0 to 9·0	D

2.2.1 When determined by the method prescribed in Appendix A of this standard, the observed extractable acids including MCPA content percent by mass of any of the samples shall not differ by more than $\pm$ 5 percent as allowable tolerances. The actual value of the technical material in the formulation shall be calculated to the two decimal places and then rounded off to the one decimal place before applying the tolerance.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in clean and dry containers made of mild steel properly and suitably lacquered from inside. For packs of 10 litres or less, containers made of tin plate properly and suitably lacquered from inside may also be used. The containers shall also comply with the general requirements as stipulated in 2 of IS : 8190 (Part II)-1976*.

3.2 Marking — The containers shall bear legibly and indelibly the following information in addition to the provisions required under the Insecticides Act and Rules:

a) Name of the material;
b) Name of the manufacturer;
c) Date of manufacture;
d) Batch number;
e) Net volume of contents;

*Requirements for packing of pesticides : Part II Liquid pesticides.

f) MCPA content, including other extractable acids, percent (*m/m*); and

g) The minimum cautionary notice worded as under:

'DANGER. KEEP OUT OF REACH OF CHILDREN. KEEP AWAY FROM FOODSTUFFS AND ANIMAL FEEDS. WASH HANDS THOROUGHLY WITH SOAP AND WATER AFTER HANDLING.'

3.2.1 Each containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 'Indian standard methods for sampling of pesticides and their formulations' (*under preparation*).

NOTE — Until the standard under preparation is published the matter shall be subject to agreement between the concerned parties.

5. TESTS

5.1 Tests shall be carried out as prescribed in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Specification for water, distilled quality (*revised*).

APPENDIX A

[*Table* 1, *Item* (i) *and Clause* 2.2.1]

DETERMINATION OF MCPA AND OTHER EXTRACTABLE ACIDS CONTENT

A-0. PRINCIPLE

A-0.1 The acids are converted to their sodium salts with sodium hydroxide, acidified with hydrochloric acid, extracted with diethyl ether, and then titrated with standard sodium hydroxide using phenolphthalein.

A-1. APPARATUS

A-1.1 Separating Funnels — 250-ml capacity.

A-1.2 Conical Flasks — 250-ml capacity.

A-1.3 Water Bath — maintained at 80° to 85°C.

A-2. REAGENTS

A-2.1 Ethanol — 95 percent (*v/v*).

A-2.2 Standard Sodium Hydroxide Solution — 0·1 N.

A-2.3 Sodium Hydroxide Solution — 2 N.

A-2.4 Hydrochloric Acid Solution — 5 N.

A-2.5 Phenolphthalein Indicator Solution — one percent (*m/v*) in 96 percent ethanol.

A-2.6 Diethyl Ether

A-3. PROCEDURE

A-3.1 Weigh accurately, 0·8 to 1·0 g of the sample and transfer, quantitatively to a separating funnel using 20 ml of sodium hydroxide solution (2 N). Shake to dissolve, and add 5 drops of phenolphthalein indicator solution, followed by sufficient hydrochloric acid to make the solution acidic. Then add 2 ml excess hydrochloric acid. Extract the solution with three successive portions of 50 ml diethyl ether and combine the extracts.

A-3.2 To check whether all the acids have been extracted, carry out a fourth extraction with 50 ml diethyl ether, and wash with not more than 10 ml of water. Transfer this extract to the conical flask. Evaporate the ether on water bath and dissolve the residue in 50 ml ethanol. Dilute the solution with 20 ml distilled water, recently boiled and cooled and titrate with standard sodium hydroxide solution using phenolphthalein.

A-3.3 If the titration of the fourth extract takes more than one drop of the standard sodium hydroxide solution, then carry out a fifth extraction, and when the conditions for complete extraction of the acids have been established, repeat the analysis. If the fourth extract does not require more than one drop of standard sodium hydroxide solution, then it may be assumed that three extractions are adequate (**A-3.1**). Discard the aqueous layer and wash the combined extracts with three successive portions of 10 ml water. Combine these water washes and extract with 15 ml diethyl ether. Discard the aqueous layer and combine the ether layers. Transfer the ether extracts to the conical flask and proceed as in **A-3.2**.

A-4. CALCULATION

A-4.1 MCPA and other extractable acids, percent by mass $= \frac{20{\cdot}06 \times N \times V}{M}$

where

N = normality of the standard sodium hydroxide solution,

V = volume in ml of standard sodium hydroxide solution used, and

M = mass in g of the sample taken for the test.

APPENDIX B

[*Table* 1, *Item* (ii)]

DETERMINATION OF FREE PHENOLS

B-0. PRINCIPLE

B-0.1 The absorbance of an ethanolic-ammonia solution of the sample is determined, after adding 4-aminophenazone and potassium ferricyanide solutions.

B-1. REAGENTS

B-1.1 Ammonia Solution — 0·05 N.

B-1.2 Ethanol

B-1.3 Solution *A* — Dissolve 100 mg 4-chloro-2-methylphenol in 10 ml acetene and dilute to one litre with distilled water. One ml of this solution contains 100 μg phenol.

B-1.4 Solution *B* — Dissolve, 0·5 g pure MCPA, free from phenols in 50 ml of ethanol, add 90 ml of 0·05 N ammonia solution and then dilute to one litre with distilled water.

B-1.5 4-Aminophenazone Hydrochloride Solution — 0·2 percent (*m/v*) aqueous solution.

B-1.6 Potassium Ferricyanide Solution — 0·4 percent, aqueous solution, freshly prepared.

B-2. PROCEDURE

B-2.1 Calibration — With a microburette, transfer 0·2, 0·4, 0·5, 0·6, 0·8, 1·0, and 1·2 ml of Solution *A* into 7 stoppered measuring cylinder and make up the volume in each to 10 ml with Solution *B*. Pipette 5 ml ammonia solution into each, mix the contents, add 5 ml of 4-aminophenazone hydrochloride solution, and mix again. Finally, add 5 ml potassium ferricyanide solution, shake vigorously for 1 minute, and after 5 to 10 minutes measure the absorbance in a 1-cm cell using distilled water in the reference cell. Determine a blank on the reagents by taking 10 ml of Solution *B* and treating with ammonia solution, 4-aminophenazone hydrochloride and potassium ferricyanide solutions as above. Subtract the 'blank' from the reading obtained on the phenol solution. Prepare a calibration graph plotting ml of Solution *A* against absorbance.

B-2.2 Determination of Free Phenols in the Sample — Weigh accurately sufficient sample to contain about 0·5 g of MCPA. Dissolve in 50 ml ethanol in the volumetric flask, and add 90 ml ammonia solution, and make up to one litre with distilled water. Pipette 10 ml of this solution into a stoppered measuring cylinder and add, in turn 5 ml ammonia, 5 ml 4-aminophenazone hydrochloride, and 5 ml potassium ferricyanide solutions, shaking after each addition Continue shaking for about 1 minute and measure the absorbance after 5 to 10 minutes. Prepare a blank as given under **B-2.1** and deduct it from the value obtained with the sample. Read the number of ml of Solution *A* equivalent to the absorbance found.

B-3. CALCULATION

B-3.1 Free phenols (expressed as 4-chloro-2-methylphenol) = $\frac{X}{M}$ percent by mass

where

X = number of ml of solution *A* equivalent to the absorbance found, and

M = mass in g of the sample taken for the test.

APPENDIX C

[*Table* 1, *Item* (iii)]

DETERMINATION OF THE COARSE MATERIAL INSOLUBLE IN WATER

C-1. PROCEDURE

C-1.1 Weigh accurately, 20 g of the sample, mix with 100 ml of water in the stoppered measuring cylinder and shake the mixture for 10 minutes. Pour the solution through a 150-micron IS Sieve (*see* IS : 460-1962*), washing out any residue from the measuring cylinder with water on to the sieve. Wash the residue on the sieve several times with distilled water and allow to drain. Brush the residue remaining on the sieve on to the glazed paper and then to the tared weighing bottle. Dry the weighing bottle and residue at 100°C, cool and reweigh. Calculate the percent by mass of coarse material insoluble in water.

APPENDIX D

[*Table* 1, *Item* (iv)]

DETERMINATION OF HYDROGEN ION CONCENTRATION (*p*H)

D-1. APPARATUS

D-1.1 ***p*H Meter** — Standardized against buffer of *p*H 7·0.

D-2. REAGENTS

D-2.1 Water — Redistilled from all acid resistance glass apparatus, and boiled to expel all carbon-dioxide just before use.

D-2.2 Phosphate Buffer Solution — 0·025 M. Dissolve 3·402 g of potassium hydrogen phosphate and 3·549 g of sodium hydrogen phosphate in water.

D-3. PROCEDURE

D-3.1 Take 25 ml of the sample in a small beaker. Determine the *p*H of this solution using the *p*H meter.

*Specification for test sieves (*revised*).

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Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 1 kg.1 m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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